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Indoor Air Sources: Using Small Environmental Test Chambers to Characterize Organic Emissions from Indoor Materials and Products

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INDOOR AIR SOURCES: USING SMALL ENVIRONMENTAL TEST CHAMBERS TO CHARACTERIZE ORGANIC EMISSIONS FROM INDOOR MATERIALS AND PRODUCTS

by

Bruce A. Tichenor
U. S. Environmental Protection Agency
Air and Energy Engineering Research Laboratory
Indoor Air Branch
Research Triangle Park, NC 27711

AIR AND ENERGY ENGINEERING RESEARCH LABORATORY
OFFICE OF RESEARCH AND DEVELOPMENT
U.S. ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

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PREFACE

In October 1986, Congress passed the Superfund Amendments and Reauthorization Act (SARA, PL 99-499) that includes Title IV - The Radon Gas and Indoor Air Quality Research Act. This Act directs that the Environmental Protection Agency (EPA) undertake a comprehensive indoor air research program with the ultimate goal of providing information on indoor air pollution to the public.

EPA publishes the results of its indoor air research activities in the INDOOR AIR technical report series. The series consists of scientific and technical reports covering five subject categories: Sources, Measurement, Health, Assessment, and Control. These reflect the organization of the Indoor Air Research Program within EPA's Office of Research and Development:

A. Sources of Indoor Air Pollution
B. Building Diagnosis and Measurement Methods
C. Health Effects
D. Exposure and Risk Assessment
E. Building Systems and Indoor Air Quality Control Options.

Research program requirements under Superfund Title IV are specific. They include characterizing and monitoring the sources and levels of indoor air pollution; developing instruments to collect indoor air quality data; and studying high risk building types. The statute also requires research on the effects of indoor air pollution on human health. Additional research is required to develop mitigation measures to prevent or abate indoor air pollution; and to develop methods to both assess the potential for soil gas contamination of new construction, and examine design measures to avoid indoor air pollution.

EPA is directed to undertake this comprehensive research and development effort not only through in-house work but also in coordination with other Federal agencies, state and local governments, and private sector organizations having an interest in indoor air pollution.

For each of its technical reports the Office of Research and Development publishes a Project Summary. A Project Summary is a short synopsis of the key research findings of a research report, and is a means of informing the public about reports available from the National Technical Information Service and not printed for outside distribution. Project Summaries for all reports in the INDOOR AIR series are available from:

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SECTION 1

INTRODUCTION

A. SCOPE

The use of small environmental test chambers to develop emission characteristics of indoor materials and products is still evolving. Modifications and variations in equipment, testing procedures, and data analysis are made as the work in the area progresses. Until the interested parties agree upon standard testing protocols, differences in approach will occur. The purpose of this report is to provide assistance by describing equipment and techniques suitable for determining organic emissions from indoor materials. Specific examples are provided to illustrate existing approaches; these examples are not intended to inhibit alternative approaches or techniques.

Small chambers have obvious limitations. Normally, only samples of larger materials (e.g., carpet) can be tested. Small chambers may not be applicable for testing complete assemblages (e.g., furniture). Small chambers are also inappropriate for testing combustion devices (e.g., kerosene heaters) or activities (e.g., use of aerosol spray products). For some products, small chamber testing may provide only a portion of the emission profile of interest. For example, the rate of emissions from the application of high solvent materials (e.g., paints, waxes) via brushing, spraying, rolling, etc. is generally higher than the rate during the drying process. Small chamber testing cannot be used to evaluate the application phase of the coating process.

The report does not provide specific guidance for determining emissions of formaldehyde from pressed wood products, since large chamber testing methods for such emissions are well developed and widely used (Myers, 1984). It is possible, however, that the guide could be used to support alternative testing methods.

While the ultimate purpose of any evaluation of indoor materials is to determine whether the emissions can contribute to health or comfort problems, this report is applicable only to the determination of the emissions themselves. The effect of the emissions (e.g., toxicity) is beyond the scope of the report.

Within the context of the limitations discussed above, the purpose of this report is to describe the methods and procedures for determining organic emission rates from indoor materials/products using small environmental test chambers. The techniques described are useful for both routine product testing by manufacturers and testing laboratories and for more rigorous evaluation by Indoor Air Quality (IAQ) researchers.

B. TESTING OBJECTIVES

The use of small chambers to evaluate organic emissions from indoor materials has several objectives:

- develop techniques for screening of products for organic emissions;
- determine the effect of environmental variables (i.e., temperature, humidity, air exchange) on emission rates;
- rank various products and product types with respect to their emissions profiles (e.g., emission factors, specific organic compounds emitted);
- provide compound-specific data on various organic sources to guide field studies and assist in evaluating indoor air quality in buildings;
- provide emissions data for the development and verification of models used to predict indoor concentrations of organic compounds;
- develop data useful to manufacturers and builders for assessing product emissions and developing control options or improved products.

C. MASS TRANSFER CONSIDERATIONS

Small chamber evaluation of emissions from indoor materials requires consideration of the relevant mass transfer processes. Three fundamental processes control the rate of emissions of organic vapors from indoor materials: 1) evaporative mass transfer from the surface of the material to the overlying air, 2) desorption of adsorbed compounds, and 3) diffusion within the material.

Evaporative Mass Transfer

The evaporative mass transfer of a given organic compound from the surface of the material to the overlying air can be expressed as:

$$E = k_m (VP_s - VP_a) \qquad (1)$$

Where, E = Emission rate, mg/hr
k_m = Mass transfer coefficient, mg/mm Hg-hr
VP_s = Vapor pressure at the surface of the material, mm Hg
VP_a = Vapor pressure in the air above the surface, mm Hg

Thus, the emission rate is proportional to the difference in vapor pressure between the surface and the overlying air. Since the vapor pressure is directly related to the concentration, the emission rate is proportional to the difference in concentration between the surface and the overlying air. The mass transfer

coefficient is a function of the diffusion coefficient (in air) for the specific compound of interest, the level of turbulence in the boundary layer above the surface of the material, and the thickness of the boundary layer.

Desorption

The desorption rate of compounds adsorbed on materials can be determined by the retention time (or average residence time) of an adsorbed molecule (Levine, 1978):

$$\tau = \tau_o \, e^{-Q/RT} \qquad (2)$$

Where, τ = Retention time, sec
τ_o = Constant with a typical value from 10^{-12} to 10^{-15} sec
Q = Molar enthalpy change for adsorption or adsorption energy, cal/mole
R = Gas constant, 1.987 cal/mole-K
T = Temperature, K

The larger the retention time, the slower the rate of desorption.

Diffusion Within the Material

The diffusion mass transfer within the material is a function of the diffusion coefficient (or diffusivity) of the specific compound. The diffusion coefficient of a given compound within a given material is a function of the compound's physical properties (e.g., molecular weight, size), temperature, and the structure of the material within which the diffusion is occurring. The diffusivity of an individual compound in a mixture is also affected by the composition of the mixture.

Variables Affecting Mass Transfer

While a detailed discussion of mass transfer theory is beyond the scope of this guide, it is necessary to examine the critical variables affecting mass transfer within the context of small chamber testing:

Temperature--

Temperature affects the vapor pressure, desorption rate, and diffusion coefficients of the organic compounds. Thus, temperature impacts both the mass transfer from the surface (whether by evaporation or desorption) and the diffusion mass transfer within the material. Increases in temperature cause increases in the emissions due to all three mass transfer processes.

Air Exchange Rate--

Air exchange rate (ACH or hr^{-1}) is defined as the volume of outdoor air that enters the indoor environment in 1 hour divided by the volume of the indoor space. The air exchange rate indicates the amount of dilution and flushing that occurs in indoor environments. The higher the air exchange rate, the

greater the dilution and the lower the concentration. If the concentration at the surface is unchanged, a lower concentration in the air increases the evaporative mass transfer by increasing the difference in concentration between the surface and the overlying air.

Air Velocity--

The mass transfer coefficient (k_m) is affected by the velocity in the boundary layer above the surface and the level of turbulence. Generally, the higher the velocity and the higher the level of turbulence, the greater the mass transfer coefficient. In a practical sense, above a certain velocity and level of turbulence, the resistance to mass transfer in the boundary layer is minimized (i.e., the mass transfer coefficient reaches its maximum value). In chamber testing, some investigators prefer to use velocities high enough to minimize the mass transfer resistance at the surface. For example, air velocities of 0.3 to 0.5 m/sec have been used in evaluating formaldehyde emissions from wood products. Such velocities are higher than those observed in normal residential environments by Matthews, et al. (1987), where in six houses they observed velocities with a mean of 0.07 m/sec and a median of 0.05 m/sec. Thus, other investigators prefer to keep the velocities in the range normally found indoors. In either case, an understanding of the effect of velocity on the emission rate is needed in interpreting small chamber emissions data.

D. USE OF THE RESULTS

It is emphasized that small chamber evaluations are used to determine source emission rates. These rates are then used in appropriate IAQ models to predict indoor concentration of the compounds emitted from the tested material. The concentrations observed in the chambers themselves should not be used as a substitute for concentrations expected in full-scale indoor environments.

SECTION 2

FACILITIES AND EQUIPMENT

A facility designed and operated to determine organic emission rates from building materials and consumer products found indoors should contain the following: test chambers, a clean air generation system, monitoring and control systems, sample collection and analysis equipment, and standards generation and calibration systems. Figure 1 is a schematic showing an example system with two test chambers.

A. ENVIRONMENTAL TEST CHAMBERS

Small environmental test chambers are designed to permit the testing of samples of various types of building materials and consumer products. They can range in size from a few liters to 5 m^3. Generally, chambers of more than 5 m^3 are considered "large." Large chambers permit the testing of complete assemblages (e.g., furniture); they may also be used to evaluate activities (e.g., spray painting). For the purpose of this guide, small chambers are assumed to be used to test samples of larger materials and products, as opposed to full scale materials or processes.

Chamber Construction

The test chambers should have non-adsorbent, chemically inert, smooth interior surfaces. Care must be taken in their construction to avoid the use of caulks and adhesives that emit or adsorb volatile organic compounds. Electropolished stainless steel and glass are common interior surfaces. The chamber must have an access door with airtight, non-adsorbent seals. The chambers must be fitted with inlet and outlet ports for air flow. Ports for temperature and humidity probes may also be required. Ports for sample collection are needed only if the sampling is not conducted in the outlet air, although sampling in the outlet is the preferred technique.

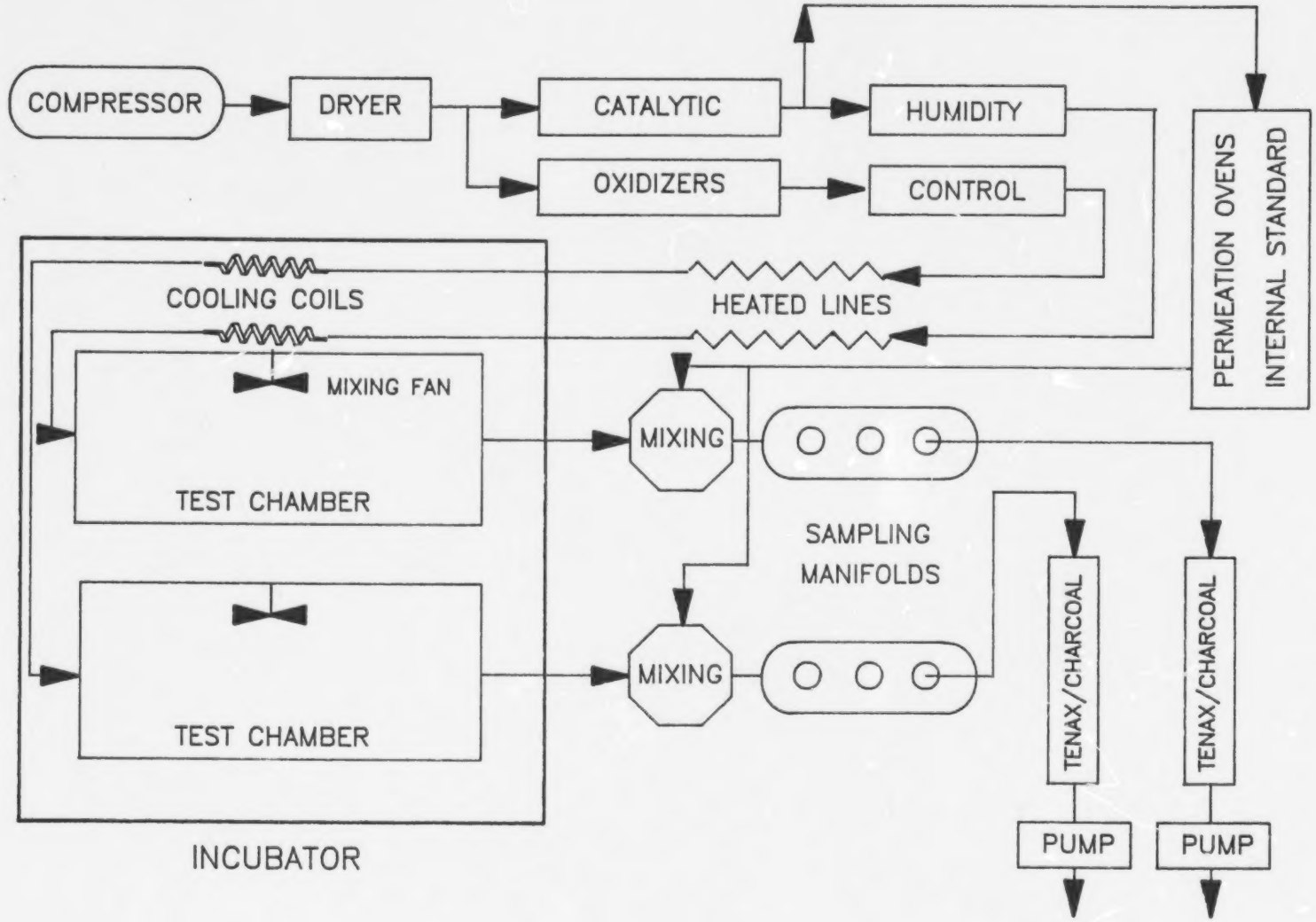

Figure 1. Schematic of example small chamber test facility.

Internal Mixing

The chambers should be designed to ensure adequate mixing of the chamber air. Low speed mixing fans or multi-port inlet and outlet diffusers are two techniques that have been used successfully. One approach for determining if the chamber air is adequately mixed is to blend a tracer gas (e.g., SF_6) with the inlet air at constant concentration and flow and measure the concentration in the chamber outlet over time. The chamber concentration vs. time plot is then compared to the theoretical curve for a completely mixed chamber:

$$C = C_o(1 - e^{-Nt}) \qquad (3)$$

Where, C = Chamber concentration, mg/m^3
C_o = Inlet concentration, mg/m^3
N = Air exchange rate, hr^{-1}; $N = Q/V$, where Q = Flow rate through chamber, m^3/hr, and V = Chamber volume, m^3
t = Time, hr

If the measured data closely follow the theoretical curve, the chamber is well mixed. When the measured data lie above the theoretical curve, short circuiting of the flow is occurring and the chamber air is not well mixed. Short circuiting is probably caused by poor placement of the air inlet and/or outlet ports. If the measured data fall below the theoretical curve, some of the tracer gas may be adsorbing on the chamber surfaces, the chamber may be leaking, or incomplete mixing may be occurring. Tests to determine the adequacy of mixing should be conducted not only in an empty chamber, but also with inert substrates of the types of samples to be tested to ensure that placement of the samples in the chamber will not result in inadequate mixing.

Quantitative guidance on the mixing is unavailable. One method might be to "force" the measured data through the theoretical curve using the chamber volume (V) as a variable. One could then compare the actual chamber volume to the "apparent" chamber volume based on the curve fit. A difference of >10% between the actual and "apparent" volumes might be considered unacceptable.

Surface Velocity

As discussed in Section 1.C, the velocity near the surface of the material being tested can affect the mass transfer coefficient. If it is desired to maximize the mass transfer in the boundary layer, one should use a relatively high velocity (e.g., >0.3 m/sec). This will require the use of a fan to direct the flow along the surface of the material. If the test objectives require low velocities more representative of indoor environments, care should be taken to ensure that the technique used to promote mixing in the chambers does not cause excessive air velocities (e.g., >0.05 m/sec) at the surface of the source to be tested.

Temperature Control

Temperature can be controlled by placing the test chambers in incubator cabinets or other controllable constant temperature environments.

Lights

Small chambers are normally operated without lights. If the effect of lighting on emissions is to be determined, appropriate interior illumination should be provided. If lighting is used, care should be taken to avoid heating of the chamber interior.

Clean Air Generation System

Clean air must be generated and delivered to the chambers. A typical clean air system might use an oilless compressor drawing in ambient air followed by removal of moisture (e.g., using a membrane dryer) and trace organics (e.g., by catalytic oxidation units). Other options include gas cylinders or charcoal filtered outdoor or laboratory air. The amount of air flow required should be calculated before a decision is reached on the supply system. The required purity of the air must also be determined based on the type of samples to be evaluated.

Humidity Control

Humidity of the chamber air is controlled by adding deionized (or HPLC grade distilled) water to the air stream. Injection by syringe pumps followed by heating to vaporize the water can achieve desired humidity levels, although syringe pumps are prone to breakdown during prolonged, continuous use. Other types of pumps (e.g., HPLC) might also provide sufficient accuracy. Humidification can also be accomplished by bubbling a portion of the airstream through deionized water at a controlled temperature (e.g., in a water bath). Coiled lines inside the constant temperature environment (e.g., incubator) can be used for inlet temperature equilibration before delivery to the test chambers.

B. ENVIRONMENTAL MEASUREMENT AND CONTROL

Measurement and control are required for air flow, temperature, and humidity. Air flow can be automatically monitored and controlled by electronic mass flow controllers, or manual flow control (e.g., needle valve, orifice plate) and measurement (e.g., bubble meter, rotameter) can be used. Some investigators recommend that the chamber be operated very slightly above atmospheric pressure and that both atmospheric and chamber pressure be measured. Temperature control is discussed above (see Section 2.A). Temperature can be measured automatically using thermocouples or thermistors; manual dial or stem thermometers can also be used. Control of humidity depends on the humidification system employed. If liquid injection is used, water flow is controlled by the pump setting. Control of

humidity by saturated air requires temperature control of the water and flow control of the saturated air stream. Humidity can be measured by several types of sensors, including dew point detectors and thin-film capacitors. Temperature and humidity sensors should be located inside the chamber at least 5 cm from the inside wall and near the midpoint between the air inlet and outlet ports.

Microcomputer based measurement and control systems can be used to set air flow rates and monitor temperature, relative humidity, and air flow during the course of experiments. Analog signals from temperature, relative humidity, and flow sensors are converted to digital units that can be stored by a microcomputer-based system, then processed to engineering units using appropriate calibration factors. In this way, chamber environmental data can be continuously monitored, then compiled and reduced for archival storage or display with minimal operator effort. Automatic systems are also capable of certain control functions. Digital signals can be output to control valves or converted to analog signals and sent out as set point signals to mass flow controllers. A graphics overlay program can be used to show current setpoints and measured values on a system schematic displayed on the microcomputer's monitor.

While automatic systems provide enhanced data collection and control, they are also expensive and complex. The simplicity and low cost of manual systems may be preferable under many circumstances.

SECTION 3

SAMPLE COLLECTION AND ANALYSIS

Indoor sources of organic emissions vary widely in both the strength of their emissions and the type and number of compounds emitted. To fully characterize organic emissions, the sample collection/analysis system must be capable of quantitative collection and analysis of volatile, semivolatile, polar, and non-polar compounds. Any small chamber sampling and analysis technique or strategy developed must consider the emission characteristics of the specific source being evaluated. The design and operation of sample collection and analysis systems must be appropriate for the organic compounds (and their concentrations) being sampled. Such systems generally include sampling devices (e.g., syringes, pumps), sample collectors (e.g., syringes, adsorbent media, evacuated canisters), and instruments to analyze organic emissions (e.g., gas chromatographs [GCs]). The remainder of this section discusses the alternatives available for small chamber sampling and analysis of organic emissions; technical details of specific systems are not included.

A. SAMPLING DEVICES

The exhaust flow (i.e., chamber outlet) is normally used as the sampling point, although separate sampling ports in the chamber can be used. A multiport sampling manifold can be used to provide flexibility for duplicate samples. A mixing chamber between the test chamber and the manifold will permit addition and mixing of internal standard gases with the chamber air stream. Sampling ports with septums are needed if syringe sampling is to be conducted. The sampling system should be constructed of inert material (e.g., glass, stainless steel), and the system should be maintained at the same temperature as the test chambers. The exhaust from the sampling system should be ducted into a fume hood, ensuring that any hazardous chemicals emitted by the test materials are isolated from the laboratory environment.

Samples can be drawn into gastight syringes, GC sampling loops, evacuated canisters, or through sorbent cartridges using sampling pumps. Gastight syringes and closed loops are frequently used when chamber concentrations are high and sample volumes must be small to prevent overloading of the analytical instrument. Larger volume samples can be pulled through sorbent cartridges using sampling pumps. Flow rate can be controlled by an electronic mass flow controller. Experience at EPA suggests that the sampling flow rate should be limited to < 50% of the chamber flow rate to prevent perturbing the chamber flow. Valves and a vacuum gauge may be incorporated into the system to permit verification of system integrity before samples are drawn. The entire system can be connected to a programmable electronic timer to permit unattended sample collection.

B. SAMPLE COLLECTION MEDIA

If the sample is collected via syringe or closed-loop sampling, it is injected directly into a GC or other instrument for analysis. Collection in a sampling bag (e.g., Tedlar) or vessel (e.g., glass, stainless steel) allows for larger samples. For many small chamber evaluations of indoor materials, low concentrations of the compounds of interest require large volume samples and collection on an appropriate adsorbent medium is required. Several sorbent materials are available for use, singly or in combination, including activated carbon, glass beads, Ambersorb, Tenax, graphitized carbon, and XAD-2. The selection of the sorbent (or sorbent combination) depends on the compound(s) to be collected. For example, a sorbent combination of activated carbon and Tenax, in series, allows quantitative thermal desorption of compounds with boiling points below 200°C. Desorption temperatures up to 230°C are commonly used; however, Tenax artifact formation increases at increasing temperatures. Graphitized carbon sorbents can be desorbed at temperatures up to 400°C and thus are useful for sampling a much wider range of compounds than Tenax. While graphitized carbon shows promise, it is emphasized that limited data are available on its performance for the wide variety of compounds emitted indoors. XAD-2 resin can be used to collect compounds considered to be semi- or non-volatile (i.e., boiling points above 180°C). Additional details on the selection and use of sorbents can be found in a series of four EPA reports (Adams, et al., 1977; Gallant, et al., 1978; Piecewicz, et al., 1979; Harris, et al., 1982).

If sorbent collection is used, the laboratory must be equipped with appropriate storage capabilities. Airtight glass tubes or chemically inert bags are both appropriate. Flushing the storage containers with high purity nitrogen prior to use will help ensure their cleanliness. Samples should be stored in a freezer at - 20°C. If possible, sorbent samples should be desorbed and analyzed within 48 hours of collection.

When sorbents are used for sample collection, desorption and concentration is necessary. For example, a clamshell oven can be used to thermally desorb sorbent cartridges with the vapors fed to the concentrator column of a purge and trap concentrator that thermally desorbs the organic compounds to the GC column. Solvent extraction and liquid injection to the GC can also be employed. Other concentration techniques are also available, including cryotrapping.

C. ORGANIC ANALYSIS INSTRUMENTATION

A variety of analytical instruments are available for determining the concentration of the organics sampled from the chamber, with GCs being the most commonly used. GCs have a wide variety of columns available for separating organic compounds. Capillary columns are generally preferred. Several detectors can be used depending on the purpose of the test and the compounds of

interest. Mass selective detectors (MSDs) are the most versatile and can be used in the scan mode to identify unknown compounds. When used in the scan mode, an MSD has a sensitivity of about 10^{-9}g. If the MSD is being used to analyze for known compounds, it is operated in the selected ion mode where its sensitivity increases to 10^{-12}g. MSDs can be made even more sensitive by negative ionization. Flame ionization detectors (FIDs) are also widely used. They respond to a wide variety of organic compounds and have a sensitivity of 10^{-11}g. Electron capture detectors (ECDs) are used for analyzing halogenated organics and have a sensitivity of 10^{-13}g. Some compounds are not easily measured with GCs; for example, low molecular weight aldehydes require other instrumentation (e.g., HPLC or wet chemical colorimetric).

D. STANDARDS GENERATION AND SYSTEM CALIBRATION

Calibration gas may be added to the test chamber or sampling manifold from permeation ovens or gas cylinders. Calibration (or tracer) gas is added through the test chamber in tests to determine chamber mixing, check for leaks, or evaluate chamber "sink" effects. Internal standards for quality control are added at the head of the sampling system. The internal standard should not be added to the chamber due to the potential for adsorption on the material being tested. Quality control can also be achieved by spiked samples.

SECTION 4

EXPERIMENTAL DESIGN

A. TEST OBJECTIVES

The first step in designing an experiment for chamber tests of indoor materials/products is to determine the test objectives. For example, a builder or architect would be interested in emissions from a variety of materials to be used under a given set of conditions for a specific building. In this case, the experiment would be designed to handle many materials with one set of environmental conditions. A manufacturer might want to know the emissions characteristics of a single product under both normal and extreme conditions and would design a test to cover the appropriate range of environmental variables. IAQ researchers interested in the interactions among variables would use a more complex design involving ranges of several variables.

B. CRITICAL PARAMETERS

A basic experimental design for small chamber tests should include consideration of the effects of various parameters on the emission characteristics of the materials to be tested. Five variables are generally considered to be critical parameters: temperature (T), humidity (H), air exchange rate (N), product loading (L), and time (t):

Temperature

Temperature affects the vapor pressure, diffusion coefficients (diffusivity), and desorption rates of the organic compounds in the materials/products and can have a major impact on emission rates.

Humidity

Humidity has been shown to affect the emission rate of formaldehyde from particleboard and may have similar effects for other water soluble gases. Humidity can be expressed in relative (% of saturation) or absolute (g water/g air) terms.

Air Exchange Rate

Air exchange rate (ACH or hr^{-1}) is determined by the mass flow rate of clean air to the chamber divided by the chamber volume. The air exchange rate indicates the amount of dilution and flushing that occurs in indoor environments and can have a major impact on chamber concentrations.

Product Loading

Product loading is the ratio of the test specimen area to the chamber volume. This variable allows product usage in the

test chambers to correspond to normal use patterns for the same product in "full scale" environments. Studies of formaldehyde emissions have shown that the ratio of air exchange rate (N) to product loading (L) is proportional to the emission rate. Thus, N/L is often selected as a parameter in designing chamber experiments. In some cases, the configuration of the source makes product loading an inappropriate parameter. For example, studies of sealants often employ elongated beads. In this case, the configuration and length of the bead are appropriate experimental design parameters.

Product Age

Age is a critical parameter, since most materials have emission rates that vary with time. Fresh, wet solvent containing products can have emission rates that vary several orders of magnitude in a few hours; other materials such as pressed wood products may have emission rates that take several years to decay.

C. PRODUCT HISTORY

Information on the history of the material/product to be tested is useful in designing the testing program. Details of manufacture, production, or assembly may be useful in determining compounds to be emitted. Information on product age, treatment (e.g., coatings, cleaning), storage conditions (i.e., time, temperature, humidity, ventilation), and handling/transportation may provide additional insight. For example, older materials may emit at a lower rate than new materials; materials stored at high temperatures may also have lower emission rates when tested; and storage or transportation with other materials may cause adsorption of organics which will be emitted during the chamber tests.

D. TEST MATRIX

For each material tested, a test matrix is developed to allow the variables of interest to be investigated. As is normal in experimental programs of this type, the desire to collect data over an extensive parameter range is limited by cost and time constraints. To maximize the information production within available resources, a statistical consultant can be used to provide guidance on appropriate experimental designs. Table 1 is an example of a test matrix developed to evaluate the effect of several variables on emission factors.

E. RECOMMENDED TEST CONDITIONS

For routine testing of indoor materials, the following test conditions are recommended:

Temperature = 23°C;
Relative humidity = 45 - 50%;
Air exchange rate = 1.0 hr^{-1}.

TABLE 1. EXAMPLE TEST MATRIX

Test No.	Temp. (°C)	RH (%)	Airflow (*l*/min)	N (hr^{-1})	Surface Area (m^2)	L (m^2/m^3)	N/L (m/hr)
1A	23	50	1.4	0.5	0.035	0.2	2.5
1B	23	50	1.4	0.5	0.035	0.2	2.5
2A	23	50	2.8	1.0	0.035	0.2	5.0
2B	23	50	2.8	1.0	0.035	0.2	5.0
3A	23	50	5.5	2.0	0.035	0.2	10.0
3B	23	50	5.5	2.0	0.035	0.2	10.0
4A	23	50	2.8	1.0	0.070	0.4	2.5
4B	23	50	2.8	1.0	0.070	0.4	2.5
5A	35	50	1.4	0.5	0.035	0.2	2.5
5B	35	50	1.4	0.5	0.035	0.2	2.5

This test matrix covers five experimental conditions, each with two replicates (A and B). The test matrix was designed to evaluate the effect of specific parameters as follows:

a) Effect of Temperature (T) - Tests 1 and 5;

b) Effect of Air Exchange Rate (N) - Tests 1, 2, and 3;

c) Effect of Product Loading (L) - Tests 2 and 4;

d) Evaluation of Constant N/L - Tests 1 and 4.

The effect of humidity was not examined during this set of experiments. The effect of age was investigated by collecting multiple samples over the drying time of the product.

SECTION 5

EXPERIMENTAL PROCEDURES

A. EMISSIONS COMPOSITION

A preliminary evaluation of the product/material is performed to guide selection of appropriate test strategies and analytical techniques. This evaluation is conducted to obtain information on the specific compounds to be quantified. If only a single compound is to be quantified, selection of the appropriate sampling and analysis strategy is straightforward, and no further screening is needed. When a more complete characterization is desired, more information is required. An initial evaluation of the composition of the emissions expected from a source can be conducted by surveying available information, including: a) reports or papers on previous studies of the source, b) ingredients listed on the product label, c) Material Safety Data Sheets (MSDS), and d) information obtained from the manufacturer or appropriate trade organizations. Such information is usually insufficient to identify the compounds of interest, but it does provide some guidance in what compounds to look for. Another problem is that the compounds emitted from the source may be formed during the use of the product or material and will not be listed as ingredients. Therefore, further analyses are required, and testing must be conducted to determine the actual compounds being emitted. One technique involves headspace analysis of the source emissions.

B. HEADSPACE ANALYSIS

The process of identifying the organic compounds present in the "headspace" or air above the material is termed "headspace analysis." Both static (i.e., closed container) and flow-through headspace analyses are used. One method of conducting a headspace analysis is to place a sample of material in a small (e.g., 1 *l* or less) container lined with inert material. For materials with high emission rates of organic compounds, the quantity of volatile organic material in the sealed (i.e., static) headspace over a 0.1 to 0.25 g sample may be more than enough to meet the detection limit requirements of an MSD operated in the scan mode or other detectors. Low emission materials, such as carpet, may require a different approach. A purge gas (e.g., nitrogen) can be pulled over the material (i.e., flow-through) and collected on a sorbent trap. Sufficient material and sampling time must be used to accumulate components to a level adequate for detection by the MSD or other detector. While headspace analyses are normally conducted at ambient temperature (e.g., 23°C) and atmospheric pressure, it may be necessary to increase the temperature (and thus the emissions) or collect a larger sample if insufficient material is collected for the detector being used.

The headspace components are usually identified by gas chromatography coupled with a mass selective detector (GC/MS) operated in the scan mode, although other detectors can be used if sufficient information is available on the retention times for all compounds of interest for a given GC column, gas flow, and temperature program. Use of several sampling and analytical approaches may be necessary to characterize the spectrum of compounds present in the headspace of a material. Techniques applied depend upon such factors as polarity, solubility, and boiling points of the compounds emitted. A variety of sorbent materials are available (see Section 4.3). Once the sample is collected, appropriate techniques (e.g., thermal desorption or solvent extraction) are used to remove the organics from the sorbent. Methods for injecting the sample into the GC will depend on the sample phase (vapor or liquid) and on the specific equipment available.

If different instruments are used for the headspace analysis and chamber testing, the GC column, gas flow, and temperature program used in both instruments should be the same so the retention times for the compounds selected for quantification will be known.

Based on the study objectives, some (or all) of the compounds identified in the headspace analysis are selected for measurement and quantification in subsequent chamber tests. Criteria for selection of compounds may include: major peaks in the gas chromatograph; known carcinogen, toxicant, or irritant; and low odor threshold.

While the headspace analysis provides useful information on the direct emissions from the material or product of interest, it does not ensure that all emissions will be identified. Sampling and analysis techniques may be insufficient, or compounds not found in the headspace may be emitted later due to being formed in the drying process or by interactions with the substrate.

C. CHAMBER TESTING

Chamber testing requires a preparation phase as well as a testing phase. The preparation stage begins with development of the test plan that specifies environmental conditions for each test (see Section 4 - Experimental Design), method of application of the material, conditioning period, and methods of sample collection and analysis. Development of the test plan is followed by calibration of environmental control and measurement systems, sample collection and concentration devices, and analytical systems as specified in the QA Plan. At this stage the information from the GC/MS headspace analysis is evaluated to provide guidance in selection of analytical columns, detectors, sample collection media, and an appropriate internal standard.

Internal Standard

The internal standard, an organic compound added at a known rate to the chamber exhaust, must meet several criteria:

a) it must be a readily available material (i.e., suitable for use in a permeation tube or available in a gas cylinder);

b) it must have a retention time on the analytical column that does not overlap other compounds emitted by the material;

c) it must be able to be quantitatively collected and recovered from the sample collection media used during the testing.

Also, it is desirable that the internal standard be inexpensive and have low toxicity.

Chamber Preparation

Prior to actual testing, chambers are cleaned by scrubbing the inner surfaces with an alkaline detergent followed by thorough rinsing with tap water. Deionized water is used as a final rinse. Chambers are then dried, placed in position in the temperature controlled environment, and purged at test conditions. Chamber background is monitored to ensure that background contamination is within QA limits. At this point, the chamber conditions are at test setpoints of flow and relative humidity, all analytical systems have been calibrated, the QC system has been developed, and the internal standard has been selected. A chamber background sample is then taken to quantify any contribution of organic compounds from the clean air system and/or the empty chamber. In addition, any substrate materials, such as wood, that will be used during the tests must be included to account for actual background. Once all the preparatory steps have been completed, testing of the selected material/product can commence.

Specimen Preparation

The types of test specimens used in the chambers vary according to the material or product being tested. Solid materials are tested "as is." If emissions from edges may differ from the normally exposed surface, the edges should be sealed. For example, particleboard specimens can have their edges sealed with sodium silicate to eliminate excessively high edge emissions. "Wet" materials are applied to a solid substrate. For example, a wood stain would be applied to a board, or a vinyl floor wax, to floor tile. As noted above, the uncoated substrate should be placed in the chamber during background tests to determine the magnitude of its organic emissions. Also, substrate edge effects should be eliminated by edge sealing. Wet materials are applied to the substrate outside the chamber and

placed in the chamber shortly thereafter. The start of the test (time = 0) is set when the door to the chamber is closed. As discussed in Section 1.A. Scope, small chambers are not suitable for evaluating the application phase of wet material use. Thus, emissions from the earliest portion of the drying cycle (i.e., from application until placement in the chamber) will not be measured. The time between application and the start of the test should be less than 10 minutes; the time of application and the test start time should both be recorded.

Specimen Conditioning

In some cases, emissions data are desired on later stages of a material/product life cycle (e.g., several months after a coating has been applied). In these cases, the specimen must be conditioned prior to testing. Conditioning should occur under the same environmental parameters (temperature, humidity, air exchange rate, and product loading) as those used for chamber tests. If this is not possible, the conditioning environmental parameters should be well documented. Ideally, the sample should be conditioned over its complete life cycle up to the time of testing. If this is not possible, conditioning should be conducted for a period of time sufficient to allow the emissions to equilibrate to the test conditions (e.g., 1 to 2 weeks).

Specimen Contamination

Care should be taken in testing materials which have been used or stored with other materials. In such cases, the material of interest could have acted as a "sink" and adsorbed organics from the other materials. Subsequent testing could provide emissions data which represent the re-emission of the adsorbed compounds rather than emissions from the original material.

D. SAMPLING AND ANALYSIS

Sampling

Collection of a representative sample of chamber effluent requires the use of a sampling strategy that is appropriate to the ranges of volatilities of the compounds present. The information obtained from the GC/MS headspace analysis can be used to select appropriate sample collection and concentration media. As discussed above, the sampling method can range from syringe/pump sampling to adsorption on various sorbent media.

Sampling techniques and sampling times must also be appropriate to the concentrations of compounds in the chamber air stream over time.

For constant emission rate sources, the sampling times are not critical since the chamber concentration will reach a constant equilibrium value. A minimum of three samples should be taken after the time required to reach 99.9% of the equilibrium value. Equation 3 can be rearranged and used to calculate this

time, based on the air exchange rate, N (i.e., $0.999 = 1 - e^{-Nt}$; $e^{-Nt} = 0.001$; $Nt = 6.9$; $t = 6.9/N$). Thus, at an air exchange rate (N) of 1 hr^{-1}, it takes 6.9 hours for the chamber concentration to reach 99.9% of its equilibrium value when a constant emission source is placed in the chamber at time = 0; for N = 0.5, it would take 13.8 hours, etc.

When testing wet materials such as glues, waxes, and wood finishes, chamber concentrations may change by orders of magnitude over a period of minutes, as shown in Figure 2. Accurate description of chamber concentration with time may require sampling very frequently or use of a continuous or semi-continuous monitor. A combination of both techniques is the most effective way to characterize rapidly changing emissions. The concentration of individual compounds varies as the material ages. In some cases, compounds not detected in the headspace or in the first few hours of testing may become the major emission component. Therefore, a total hydrocarbon monitor can be effective in tracking rapidly changing concentrations but may provide an incomplete qualitative picture.

It is important, therefore, to monitor changes in the emission profile as the material dries. The sampling strategy should provide a means to collect approximately the same mass in each sample. Thus, the sample volume is an important consideration. When chamber concentrations are high, sample volume must be kept low to avoid breakthrough in the collection trap or overloading of the concentrator column of a purge and trap device. Sample volumes less than 1 *l* can be drawn directly by gastight syringes, then injected through a heated port to a clean air stream flowing through sampling cartridges. Much smaller samples (e.g., 1 cc) can be injected directly into the GC. Larger volume samples are taken by pulling the chamber air stream through sample cartridges as described above. Since the flow through the cartridges is constant, increasing the sampling time will increase the sample volume. It may be necessary to conduct trial runs to develop a sampling strategy.

Extreme care must be employed in handling the sample cartridges to avoid contamination. One technique is to immediately place the cartridge in a sealed inert (e.g., Teflon) bag that has been purged with nitrogen. Glass tubes with airtight fittings can also be used.

Analysis

The analysis technique depends on the sampling strategy and adsorbent media employed. Methods of introducing the sample to the GC include direct injection, thermal desorption followed by purge and trap concentration, and solvent extraction followed by liquid injection.

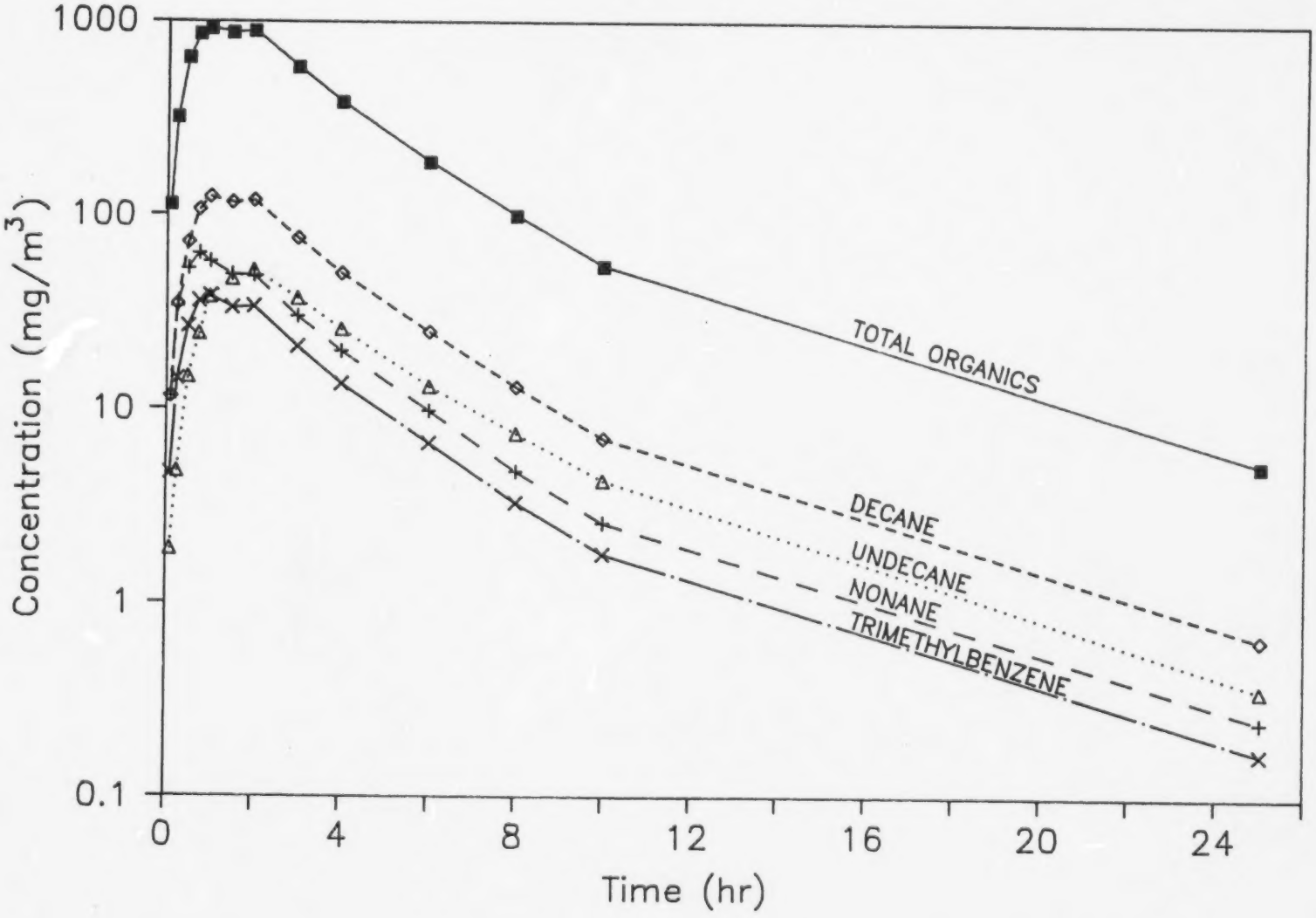

Figure 2. Chamber concentration vs. time -- wood stain

SECTION 6

DATA ANALYSIS

Data reduction and analysis is a multistep process. Electronic spreadsheets can be used to reduce and compile the environmental and chemical analysis data with minimal data entry steps. Chamber concentration data are used in various models to produce estimates of material/product emission rates.

A. ENVIRONMENTAL DATA

Environmental data (i.e., temperature, relative humidity, flow rate) can be recorded manually or automatically stored (e.g., on floppy disks) by a PC based system. Summary statistics that describe the environmental condition "setpoints" and the actual values achieved (including variability) can be computed, and a data summary sheet prepared (see Table 2).

TABLE 2. EXAMPLE ENVIRONMENTAL DATA SUMMARY

Test ID Number: PWF10

Material: Polyurethane Wood Finish

Sample Size: Weight = 2.39 g Area = 347 cm^2

Chamber No.: 1 Chamber Volume: 0.166 m^3

Material Loading (L): 0.21 m^2/m^3

Start Date	Start Time	End Date	End Time
6/16/87	1105	6/19/87	1300

Chamber Environmental Parameters

Parameter	Setpoint	Average	Standard Deviation	Maximum/ Minimum
Temp (°C)	35.0	34.91	0.18	35.4/34.5
RH (%)	50.0	54.25	1.57	60.4/45.2
Flow (*l*/min)	2.8	2.72	0.01	2.86/2.67

B. GAS CHROMATOGRAPHY DATA

GCs (including GC/MS) are interfaced to computing integrators (or PC-based chromatographic data analysis systems) for plotting of the chromatograms and computation of the areas of peaks obtained. The data output is printed on paper as an analog chromatogram plus a summary report. The data can also be stored on magnetic media for future review or reprocessing.

The environmental information and the GC analysis results are combined to give chamber concentrations for individual compounds and total organics. In calculating concentrations, the following factors are considered:

* Gas chromatographic system background (Includes sorbent blank for sampling cartridge and purge and trap concentrator);

* Chamber background (Determined from analysis of 10 to 20 *l* sample of chamber background, including substrate);

* Elapsed time (Period of time in minutes from start of test to midpoint of sampling period);

* Flow rate of the airstream carrying the internal standard;

* Mass of internal standard added and mass observed providing percent recovery;

* Mass observed for individual selected organic compounds;

* An estimate of the total organics reported as a given compound (e.g., toluene);

* Sampling duration and flow rate; and

* Test chamber flow rate.

Chamber concentrations for total organics and individual compounds for each sample are calculated using a multi-step process:

* Data may be normalized to the recovery of the internal standard by multiplying the measured mass by the reciprocal of percent recovery of the internal standard. For example, if the percent recovery was 95%, the multiplier would be 1/0.95 = 1.053.

* Normalized mass is adjusted for system background and chamber background.

* The adjusted mass is divided by sample volume to generate sampling manifold concentration data.

* Finally, chamber concentration is calculated by multiplying the sampling manifold concentration data by the ratio of flow out of the chamber plus standard addition flow divided by flow out. This compensates for dilution of the chamber effluent with the internal standard flow.

Chamber concentration data coupled with sample size and chamber air exchange rate are then used to estimate emission factors, as discussed below.

C. EMISSION FACTOR CALCULATIONS

Emission factors for organics from indoor materials are usually expressed in terms of mass/area-time. In some cases, emission factors are reported as mass/mass-time or, in the case of caulk beads, mass/length-time, when a standard bead diameter is used. (For convenience, the remainder of this section shall use emission factor units of mg/m^2-hr.) They are calculated for individual organic compounds, as well as for total measured organics. The method for calculating the emission factor depends on the type of source being tested.

Constant Emission Rate

For materials with a relatively constant emission rate over the test period, the chamber concentration will reach and maintain a constant equilibrium value. For such materials the calculation of the emission factor, when sinks are ignored, is straightforward:

$$EF = C(Q/A) \qquad (4)$$

Where, EF = Emission factor, mg/m^2-hr
C = Equilibrium chamber concentration, mg/m^3
Q = Flow through chamber, m^3/hr
A = Sample area, m^2

An equivalent expression is also used:

$$EF = C(N/L) \qquad (5)$$

Where, N = Chamber air exchange rate, hr^{-1};
L = Chamber loading, m^2/m^3

Note that N = Q/V, where V = Chamber volume, m^3.

Decreasing Emission Rate

For sources that have emission rates that decrease over the test period, a different procedure is required. The following method applies to sources with initially high emission rates that decrease with time. Most "wet" sources exhibit such behavior.

Models have been developed to analyze the results of the chamber tests to provide emission rates (Tichenor, et al., 1988). The simplest model (i.e., neglecting sink and vapor pressure effects) assumes: a) the chambers are ideal continuous-stirred tank reactors (CSTR), and b) the change in emission rate can be approximated by a first order decay, as shown in Equation 6:

$$R = R_o e^{-kt} \quad (6)$$

Where, R_o = Initial emission factor, mg/m^2-hr
k = First order rate constant, hr^{-1}
t = Time, hr

The mass balance for the chamber over a small time increment dt is:

Change in mass = Mass emitted - Mass leaving chamber

This can be expressed as:

$$VdC = AR_o e^{-kt}dt - QCdt \quad (7)$$

Equation 7 can be rearranged:

$$dC/dt + (Q/V)C = (A/V)R_o e^{-kt} \quad (8)$$

Equation 8 is a linear, non-homogeneous differential equation. Given that C = 0 when t = 0, the solution to Equation 8 is:

$$C = AR_o(e^{-kt} - e^{-Nt})/V(N - k) \quad (9)$$

Using a non-linear regression curve fit routine, implemented on a microcomputer, values of R_o and k can be obtained by fitting the concentration vs. time data from the chambers to Equation 9. In order to conduct such analyses, initial estimates of R_o and k are required. A good initial estimate of k is:

$$k = Ne^{(k-N)t_{max}} \quad (10)$$

Where, tmax is the time of maximum concentration (Cmax).

Note that Equation 10 often has two roots, one of which is k = N. The other root should be selected. Graphical, trial and error, or root trapping techniques can be used to estimate k from Equation 10.

Equation 10 is obtained by substituting C (Equation 9) into Equation 8 and setting dC/dt = 0 at t = tmax. Once an estimate of k is achieved from Equation 10, an initial estimate of R_o can be obtained from Equation 9. Figure 3 illustrates the curve fitting process for total organics data from a wood stain chamber test; the solid line is the "best fit" of Equation 9, and the data points are shown as diamonds. (Figure 3 shows only the first 10 hr of data, but the fit was made over the total test period.) Once R_o and k are determined, the value of R at any time t can be calculated from Equation 6.

It is emphasized that the methods for determining emission factors presented above are not applicable to sources that do not exhibit either constant or simple exponential decay emissions over time, and other emission models may be required. In addition, the above calculation methods do not explicitly account for several factors that may impact emission rates, including:

- the effect of chamber concentration on evaporative mass transfer, as described by Equation 1;

- the effect of adsorption to and re-emission from "sinks"; and

- the effect of diffusion within the sample substrate.

Models are being developed, based on fundamental mass transfer processes (see Section 1.C), to include consideration of these factors, but their use requires sophisticated statistical tools and analysis experience that are not generally available. As advances in source models are made, these "tools" will become more available.

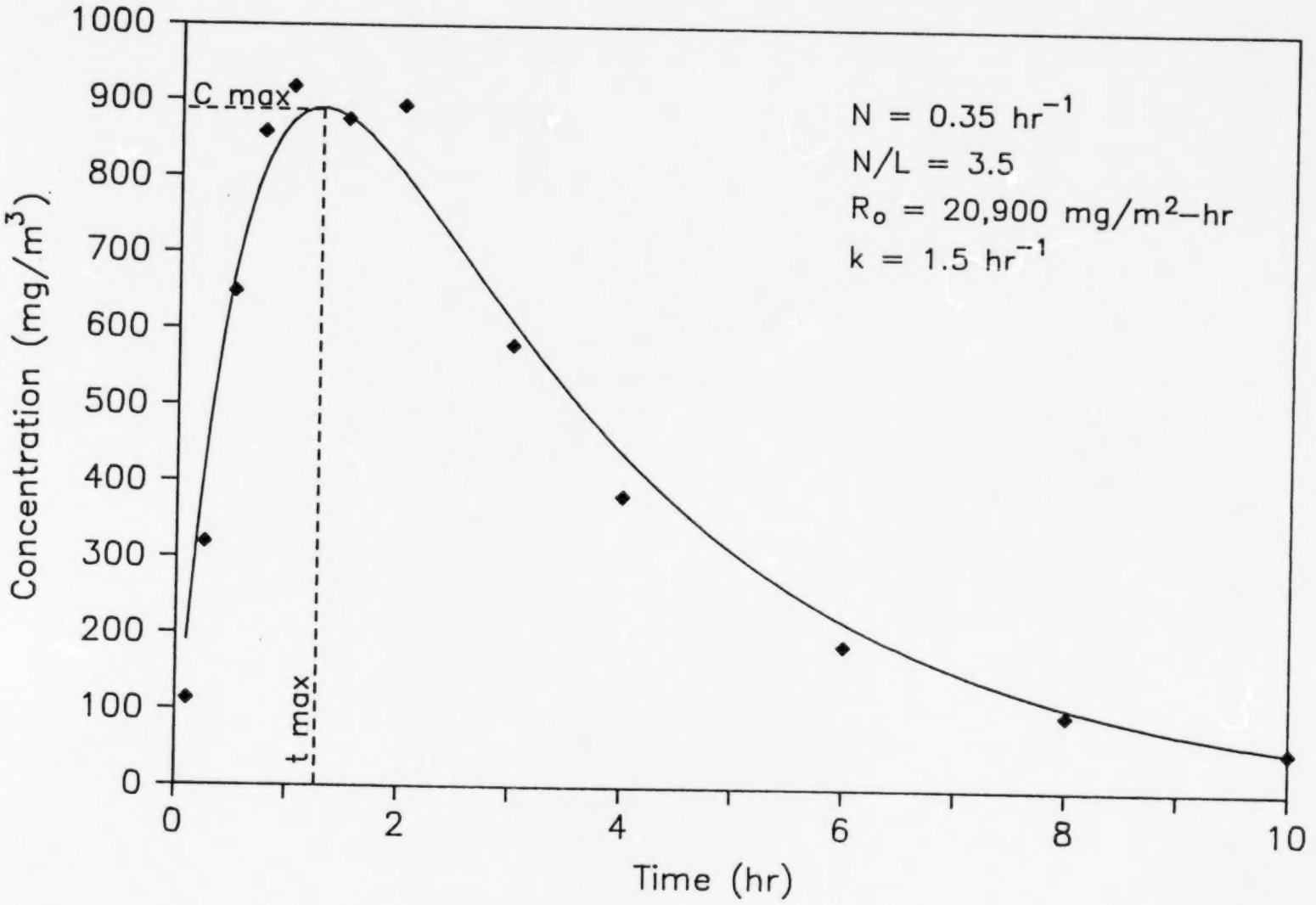

Figure 3. Example chamber concentration curve for a wet source (total organics from wood stain)

SECTION 7

QUALITY ASSURANCE/QUALITY CONTROL

Small chamber testing of organic emissions from indoor materials/products should be conducted within the framework of a Quality Assurance Project Plan (QAPP). The QAPP should contain a project description, data quality objectives/acceptance criteria, QA/QC approaches/activities, and QA/QC audits.

Project Description

A brief description should include what materials are to be tested; how the testing is to be conducted; and who is responsible for various project activities. The project experimental design (see Section 4) should contain the necessary information for this portion of the QAPP.

Data Quality Objectives/Acceptance Criteria

This section of the QAPP defines the precision, accuracy, and completeness desired for each parameter being measured. Table 3 provides an example.

QA/QC Approaches/Activities

The types of QA/QC activities that might be specified in the QAPP include establishment of a system of records/notebooks to ensure proper operation of equipment and recording of data, such as:

* Sample log to record receipt, storage, and disposition of materials;

* GC standards preparation log to document preparation of all organic compound standards;

* Permeation tube log to record weight loss data for all permeation tubes;

* Calibration logs to contain environmental systems calibration data;

* Instrument maintenance logs to document maintenance and repairs of all equipment;

* Materials testing logs in which to record all pertinent information for each test, including sample details, sample ID number, and GC run ID number;

* Sorbent cartridge cleanup/desorption log detailing thermal cleanup and QC validation of sorbent cartridges;

* Floppy disk storage log to document location and content of electronically stored data; and

TABLE 3. EXAMPLE DATA QUALITY OBJECTIVES/ACCEPTANCE CRITERIA

Parameter	Precision	Accuracy	Completeness
Temperature	±0.5°C	±0.5°C	>90%
Relative Humidity	±5.0%	±10.0%	>90%
Air Flow Rate	±1.0%	±2.0%	>90%
Substrate Area	±1.0%	-	>90%
Sample Weight*	±10.0%	-	>90%
Organic Concentration	±20% RSD**	-	>90%
Emission Rate	±20% RSD	-	>90%

* For wet samples.

** RSD = Relative Standard Deviation = (s/m)100%

Where, s = estimate of the standard deviation
m = mean

Note: Precision and accuracy are normally reported as ± 1 standard deviation unless otherwise noted. Completeness refers to the percentage of planned measurements actually conducted. For example, if 100 measurements were planned and 92 were conducted, the completeness would be 92%.

* Manuals governing operation of all equipment used by the project.

QC activities are carried out by project staff in a routine, consistent manner to provide necessary feedback in operation of all measurement systems. Such activities might include:

* Routine maintenance and calibration of systems;

* Daily recording of GC calibration accuracy and precision (i.e., control charting);

* Timely monitoring of percent recovery of the internal standard that was added to all samples;

* Collection and analysis of duplicate samples;

* QC checking of organic collection sorbent tubes; and

* Periodic analysis of audit gases supplied by an independent source.

QA/QC Audits

Finally, the QA/QC program should include periodic audits by QA personnel to evaluate compliance with QAPP protocols.

SECTION 8

REPORTING TEST RESULTS

The report of the test results should contain test objectives, facilities and equipment, experimental design, sample descriptions, experimental procedures, data analysis, results, discussion and conclusions, and QA/QC.

Test Objectives

Describe the purpose of the test program.

Facilities and Equipment

Describe the test chambers, clean air system, environmental measurement and control, sample collection (including adsorbents if used), analytical instrumentation (e.g., GC, GC/MS), and standards generation and calibration.

Experimental Design

Describe the test conditions, including temperature, humidity, air exchange rate, and material loading; include a test matrix if appropriate.

Sample Descriptions

Describe the sample(s) tested, including type of material/product, size or amount of material tested, product history, brand name (if appropriate), and sample selection process (e.g., random). For wet samples, describe the sample substrate. Also, provide information on sample conditioning, including duration and environmental conditions.

Experimental Procedures

Describe the experimental procedures used during the testing, including details of the sampling and analysis techniques and references to published methods. For wet samples, provide information on the application method.

Data Analysis

Show the methods, including appropriate models or equations, used to analyze the chamber data to produce emission factors.

Results

Provide emission factors for each type of sample tested and for each environmental condition evaluated. Emission factors can be provided for individual organic compounds and/or total organics. For sources with variable emission rates, provide appropriate rate constants.

Discussion and Conclusions

Discuss the relevance of the findings and provide conclusions. For example, describe the effect of temperature and/or air exchange rate on the emission factor.

Quality Assurance/Quality Control

Describe the Data Quality Objectives and discuss adherence to the Acceptance Criteria. This should be done for both the environmental variables and the chemical results. Provide the results of duplicate and replicate sampling, and discuss the outcome of any audits.

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The scientific and technical literature contains numerous references that report on the use of small environmental test chambers for determining emissions of organic compounds from indoor materials and products. The following references give information on approaches to small chamber testing taken by several investigators. These references are provided as suggested reading for those who wish to supplement the information contained in this guide. Further references are cited in each of these publications.

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